



# Article Analysis of the Influence of Microwave Treatment Parameters of Wood

Fernando J. R. Mascarenhas <sup>1,2,\*</sup>, Alfredo M. P. G. Dias <sup>1,2</sup> and André L. Christoforo <sup>3</sup>

- <sup>1</sup> ISISE, Department of Civil Engineering, Rua Luís Reis Santos—Pólo II, University of Coimbra (UC), 3030-788 Coimbra, Portugal
- <sup>2</sup> SerQ—Forest Innovation and Competence Centre, Rua J, N° 9, Zona Industrial da Sertã, 6100-711 Sertã, Portugal
- <sup>3</sup> Department of Civil Engineering, Federal University of São Carlos (UFSCar), Rodovia Washington Luís (SP-310), Km 235, São Carlos 13565-905, SP, Brazil
- \* Correspondence: fer.jr.resende@hotmail.com

Abstract: Microwave (MW) treatment is an effective method in the wood modification field. It has become more popular in the past decade since it enhances wood permeability, allowing a more efficient impregnation of preservative chemicals. Due to the number of parameters involved in the MW treatment of wood, multiple regression models and statistical analysis can effectively evaluate the relationship between various parameters. Therefore, this work aimed to evaluate the effects that the isolated and combined parameters of the MW treatment had on the variations of the flexural modulus of rupture (MOR) after wood specimens were MW-treated. The analyzed variables and their respective data were obtained from works on the use of MW technology for wood treatment present in the literature. Even faced with the reduced database, sufficient information was available to be used and important and accurate results were drawn. Based on the ANOVA results, wood density, initial moisture content (IMC), MW applied energy, and the product between MW power and exposure time were considered significant and could distinctly explain the reductions in the MOR values of MW-treated wood samples.

**Keywords:** microwave treatment; statistical analysis; microwave parameters; literature data; flexural strength

## 1. Introduction

Wood is one of the world's oldest construction materials. It has a wide range of applications, such as furniture and cladding material, in the construction and pulping sectors, because of its characteristics, abundance, and sustainability. As a material for construction, for instance, it has high compatibility with other building materials like concrete and steel. However, several wood species have low permeability which creates issues during the production of lumber, including prolonged drying periods, material losses after drying, and costly drying techniques [1]. Hence, developing innovative wood-based materials and technologies is critical from both scientific and industry perspectives [1]. Thus, wood industries and researchers have conducted various studies to increase wood properties, serviceability, and durability.

The procedures that have emerged as a consequence of this scientific investigation and study are called "Wood Modification Methods" [2]. Microwave (MW) treatment has presented itself as a feasible technique in the wood modification field. It has been an increasingly important topic of research interest, mainly in the past decade. Since 1945/1946, when it was invented by Percy Spence [3], MW has been applied in the heating process of food, dissolving plasma gas species, powder synthesis, satellite communications, sintering, biomass liquefaction, and enhancing the designed chemical and physical qualities of several products. It has also been used as a phytosanitary measure [4–6].



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**Copyright:** © 2023 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). Recent works have provided important information showing that MW treatment improves wood's permeability, mainly of refractory species, which allows, for example, a more effective impregnation of preservative agents [3,7–10]. Jang and Kang [11] highlight that MW technology is utilized in the wood industry to improve the effectiveness of wood drying. In addition, MW treatment is an environmentally friendly, cost-effective, and time-saving method that has been shown to significantly shorten processing times while improving drying quality [3,7,12–14]. Oloyede and Groombridge [15] demonstrated that wood samples with the same dimensions and moisture content took some days to be air-dried, several hours to be kiln-dried, and a few minutes to be MW-dried.

Eucalypt, for instance, is a wood species with low permeability, which might be challenging to be dried and impregnated with preservative chemicals [13]. The water uptake capability of small clear samples of Portuguese *Eucalyptus globulus* L. made only of heartwood enhanced by around 27% after MW drying [16]. Applying high-intensity MW treatment, the retention of acid copper chromate of MW-treated samples of *Eucalyptus tereticornis* jumped from 2.03 kg/m<sup>3</sup> to 9.72 kg/m<sup>3</sup>, a rise of more than 375% [17]. After MW treatment, the uptake of Tanalith E of MW-treated samples of Spanish *Eucalyptus globulus* L. increased by 148% compared to wood specimens with no MW treatment [13].

Samani et al. [14] found a significant increase in preservative retention and penetration of MW-treated wood specimens of *Melia composita* with increasing MW treatment duration compared to control samples. Poonia and Tripathi [8] found an improvement of two times in the retention capability of MW-treated *Pinus roxburghii* Sarg samples compared to a control group. Furthermore, Kol and Çayır [18] treated wood samples of *Picea orientalis* containing only sapwood under MW applied energy of 2158 MJ/m<sup>3</sup> after impregnating them with Tanalith E 8000. Based on this, the authors measured an increase of 61% in the rate of retention when compared to wood samples with no MW treatment [18].

In conventional wood drying, the heat is transmitted from the outer layers toward the inner of the specimen through convection, conduction, and radiation. In MW treatment, the electromagnetic waves penetrate the entire volume of the specimen, hence promoting the volumetric absorption by the wet wood specimen [19,20]. Under the conventional drying heat process, the energy is transported because of thermal gradients in heat transfer. In microwave heating, the electromagnetic energy is converted to thermal energy rather than heat transmission. It causes rapid heating across the material thickness while also reducing thermal gradients. Volumetric heating can also help to shorten drying times and save energy [19].

Oloyede and Groombridge [15] explain that when MW waves enter contact with moisture wood, the water molecules get orientated to the electromagnetic field of the MW and start to oscillate. This movement increases the wood's internal temperature, generating steam pressure that can achieve around 600 kPa in a few seconds [12,21,22]. This growing steam pressure causes varying degrees of damage to the wood cell tissue [10]. Tyloses in vessels, pit membranes in cell walls, and fragile ray cells are examples of microstructure that may be ruptured [23]. Changes in microstructure may lead to variations in porosity and pore diameter distribution, creating new pathways to make liquid transit easier. Therefore, they impact essential aspects of end-product characteristics, including permeability [23,24].

Several studies have demonstrated the chemical modifications in the conventional thermal modification process, such as the severe decomposition of hemicelluloses [25]. However, regarding MW treatment, a few studies have already investigated the chemical properties of MW-treated wood samples. Wang et al. [26] stated that the chemical structures of the three primary components of wood—cellulose, hemicellulose, and lignin—changed as a result of microwave treatment. In contrast, cellulose and lignin were reasonably stable. They only changed within a narrow range of conditions, and hemicellulose suffered substantial damage [26]. The microstructural modifications that MW treatment causes in wood cells impact not only the physical properties of wood but also its mechanical ones.

Different studies have shown that depending on the MW power and exposure time, the values of modulus of rupture (MOR) and other strength properties of MW-treated samples might have different levels of decrease [4,7,12,13,18,27]. Based on the conditions of MW treatment, Torgovnikov and Vinden [12] stated that structural changes in wood that occur after MW treatment and their impact on wood properties might be grouped into three degrees of modification, low, moderate, and high. With a low degree of modification, the authors explained that there was an increase of up to 1.5 times the permeability and minimal impact on wood's mechanical properties. The moderate degree of modification increases the permeability by a thousand times and reduces the mechanical properties of wood. Finally, the high degree of modification improves the permeability substantially, but it also reduces the mechanical properties considerably [12].

Hermoso and Vega [13] noticed reductions of approximately 19, 23, 30, 28, and 7% in the modulus of rupture, modulus of elasticity, compression strength, tensile strength, and shear strength, respectively, of small clear wood samples of *Eucalyptus globulus*, under an MW energy of 430 MJ/m<sup>3</sup>. Drying wood samples of Caribbean Pine, with a MW full power of 1600 W, Oloyede and Groombridge [15] identified a decrease of 59 and 53% in the tensile strength and modulus of elasticity, respectively.

In addition to this, experimental research has demonstrated that MW treatment might depend on several parameters, such as wood species, initial moisture content (IMC), dimension of the specimens, MW frequency, power, exposure time, and the applied energy, which is the product of MW power per exposure time divided by the volume of MW-treated samples. The works in the literature demonstrate that a significative range of IMC has been used as well as different types of wood species, which implies, for instance, various densities. Furthermore, different types and dimensions of MW ovens have been used, from household to industrial MW devices [3].

Despite there being several studies on applying MW technology for wood modification, most focus on analyzing the improvements in permeability and porosity. Few of them demonstrate the impacts that different MW powers, exposure times, and applied energies have on the mechanical properties of different wood species. These analyses are important to encourage the development of novel research projects using not only small clear wood specimens but especially wood elements with larger dimensions and structural sizes for later use with various purposes.

In this sense, MOR is the most useful wood mechanical property for solid timber applications. For instance, various studies have focused on estimating MOR values of different wood species by non-destructive tests or by empirical mathematical equations [2,28–31]. International standards grade the different wood species based on their MOR values, such as the newest version of the Brazilian ABNT NBR 7190 [32] and the European EN 338 [33].

As the factors influencing the MW treatment of wood are diverse, it is crucial to detect patterns among them, i.e., identify which of them, when combined, effectively impact mechanical properties, namely the MOR in the bending of MW-treated specimens. The variability of factors involved in the MW treatment of wood specimens might even make it difficult to effectively understand all the variables that explain the variations in the values of MOR. However, it is possible to have an adequate understanding of the phenomenon by verifying the influence (significance) of each, isolated by using correlation analyses and the combined effects of the variables by using multiple regression models and an appropriate analysis of variance.

Statistical analysis and regression models are powerful tools for quantifying the association between different parameters in various scientific fields, such as civil engineering [29,34]. In the field of conventional heat treatment, several research works have been conducted and demonstrated the correlation between the different variables involved in the thermal modification process to understand and better control the quality of the heat-treated wood [29]. Pearson's correlation coefficient is one of the most used methods to investigate the linear relationship between two variables. Moreover, statis-

tical regression models have been widely used to estimate the mechanical properties of heated treated wood specimens [29] and to analyze the impact of the combined effect of different variables.

Although MW treatment is an area of study in wood modification and processing that has become increasingly popular, more studies that evaluate the impacts on the mechanical properties are still needed, covering a greater diversity of species and applying different MW intensities (applied energy). Furthermore, there is still no study in the literature that evaluates the statistical significance of the wood density and IMC and MW parameters in the MOR values of the MW-treated wood samples and, more precisely, in the variations of MOR values in the bending of MW-treated specimens when compared to those wood samples with no MW treatment. In addition, no study explains how the combination of these parameters influences the mechanical response of MW-treated wood samples through statistical analysis.

Hence, the main objective of this study is to provide a better understanding of the effects that certain MW treatment variables have on the variation of MOR values in the bending of MW-treated wood specimens when compared with those without MW treatment by carrying out an analysis of variance, correlation analysis, and statistical regression models analysis, which evaluate the influence of combined variables. The data from experimental tests presented in the literature about MW treatment of wood were used in this work to carry out the entire analysis.

#### 2. Materials and Methods

#### 2.1. Materials

Aiming to understand the influence of some wood properties and different MW parameters on MOR values in flexural bending of MW-treated wood specimens, the most relevant research works presented in the literature were used. We tried to present the largest number of experimental works that contemplated the most complete data regarding the parameters of MW treatment and information on MOR in the bending test. For this reason, some works were not cited here, either because they did not investigate the impacts of MW treatment on mechanical properties or because they did not provide a minimum amount of information about the parameters used. Thus, these research works could not be used in this study. Even so, some of the works we used did not present information such as exposure time, and others did not bring the applied energy, for example.

Based on this, eleven different works were selected for this study. Although only 11 different works had been used from the literature, it was possible to group 37 different pieces of information for each variable. In other words, there were 37 different MW treatment programs. The initial variables to be investigated were wood species, density, initial moisture content and dimensions, MW power, frequency, applied energy and exposure time, and the average MOR values in flexural bending before and after MW treatment. Therefore, another way to interpret the 37 different MW treatment programs used is by saying that 37 different configurations of combinations using the initial variables studied were established. Different wood species with varying density values and dimensions and very different IMC were present in the database used in this work.

Because of the microstructural modifications that might occur during the MW treatment, MOR values after MW differ from those before MW modification. Most MOR values in bending after MW treatment were smaller than the initial (with no MW treatment). Based on this, the analysis based on percentage variations was used since they allow an evolutionary comparison between two values. Hence, in order to understand the influence that the previously mentioned parameters (wood species, density, IMC and dimensions, MW power, frequency, applied energy and exposure time) have on MOR of MW-treated wood specimens, the variation of MOR values,  $\Delta_{MOR}$ , (Equation (1)) was used.

$$\Delta_{MOR} = \frac{(MOR_{before\ MW\ treatment} - MOR_{after\ MW\ treatment})}{MOR_{before\ MW\ treatment}} \times 100$$
(1)

Regarding wood dimensions, 19% of the works used structural-size specimens, and 81% used small clear specimens. Concerning the MW treatment, the applied energy is an important parameter to be mentioned, and it is calculated according to Equation (2):

$$E = \frac{P \times T}{V} \tag{2}$$

where *E* is the applied energy, *P* is the MW power, *T* is the MW exposure time, and *V* is the volume of wood specimens per MW treatment run.

Of the 37 MW treatment programs, 54% had information exclusively about the *E*, and 46% had information exclusively about MW power and exposure time. From Equation (1), it is possible to notice that the applied energy depends on the MW power and exposure time. Based on that and the different sizes of wood specimens, the analyzed works were separated into three groups. Group A (Table 1) contained works that used structural-size specimens and presented information about the applied energy. Group B (Table 2) contained works that used small clear wood specimens and presented information about the applied energy but presented data about the applied energy. Group C (Table 3) contained works that used small clear wood specimens and did not present information about the applied energy but presented data about MW power and exposure time. It is important to state that each value presented for density and MOR in Tables 1–3 represents their respective average values, and they are values for 12% moisture content. The negative values of  $\Delta_{MOR}$  indicate that there was a decrease in MOR after MW treatment, and the positive values indicate an increase.

Since Groups A and B had information about the applied energy, which exclusively depends on the quantity and dimension of the MW-treated wood samples (volume) and the MW power and exposure time (Equation (1)), there was no need to include those variables in the analyses carried out here. On the other hand, the works from Group C had only the information about MW power and exposure time, which, together with the specimens' volume, are necessary to calculate the applied energy. However, most of the works of Group C only indicated the dimensions of the wood specimens. They did not present how many wood samples were treated during each MW run, and without their quantity, it was not possible to calculate the *E*. Therefore, the dimensions of the wood samples of Group C were also not included in the analysis. Hence, for Group C, the product of MW power by MW exposure time, MW Power  $\times$  MW exposure time, was considered in the analysis.

In addition, due to the depth of penetration of MW, the MW frequency is linked to the wood sample's thickness; deeper penetration is made possible by a lower frequency. MW frequencies of 2.45 GHz are used for wood samples with thicknesses up to 90 mm and 0.922 GHz for wood samples with thicknesses up to 280 mm [12]. From the literature, it was concluded that small clear wood specimens required MW frequencies of 0.922 GHz and structural-sized specimens required 2.45 GHz. Hence, the MW frequency was not studied in this work since it is already well-established.

Among the eleven wood species studied, 55% were softwood and 45% hardwood (Figure 1), and the density was the parameter used to quantify and numerically express the different wood species.

Finally, after going through the methodological selection process previously discussed, for each of the three groups the effects of the following parameters (variables) on  $\Delta_{MOR}$  were investigated. For Groups A and B, these were wood density and IMC, and MW applied energy, *E*. For Group C, this was wood density and IMC and MW Power × MW exposure time. The woods' densities used in this work ranged from 270 to 900 kg/m<sup>3</sup>, the IMC ranged from 12 to 164%, the applied energy from 290 to 2158 MJ/m<sup>3</sup>, and MW Power × MW exposure time from  $8.10 \times 10^4$  to  $1.80 \times 10^6$  W.s.

Research	Wood Species	Density (kg/m <sup>3</sup> )	Initial MC (%)	MW Power (W)	Applied Energy (MJ/m <sup>3</sup> )	MW Ex- posure Time (s)	Flexural Testing Standard	Number of Repli- cates	MOR before MW Treatment (MPa)	MOR after MW Treatment (MPa)	Δ <sub>MOR</sub> (%)
Torgovnikov and Vinden [12]	Blue gum (Eucalyptus globulus)	698	98	-	1030	-	-	-	118.00	47.35	-59.91
	Paulownia (Paulownia fortune)	270	164	-	920	-	-	-	40.55	22.20	-45.67
	Messmate (Eucalyptus obliqua)	770	91	-	594	-	-	-	134.00	102.00	-23.88
	Radiata pine (Pinus radiata)	420	34.5	-	420	-	-	-	81.00	35.50	-56.17
Torgovnikov et al. [35]	Blue gum (Eucalyptus globulus)	695	93	-	990	-	STM D143-94 (2007)	56	118.00	47.00	-60.20
Balboni et al. [7]	Red Stringybark	720	71	10,000–60,000	342	-	ASTM	-	111.00	99.00	-10.81
	(E. macrorhyncha)	720	71	10,000-60,000	320	-	- D143-94 (2009)	-	111.00	114.00	+2.70

Table 1. Group A—Wood properties, MW parameters, and MOR variation values from the literature of structural size specimens.

Table 2. Group B—Wood properties, MW parameters, and MOR variation values from the literature of small clear specimens.

Research	Wood Species	Density (kg/m <sup>3</sup> )	Initial MC (%)	MW Power (W)	Applied Energy (MJ/m <sup>3</sup> )	MW Exposure Time (s)	Flexural Testing Standard	Number of Replicates	MOR before MW Treatment (MPa)	MOR after MW Treatment (MPa)	Δ <sub>MOR</sub> (%)
Hermoso and Vega [13]		900	12	500	290			50	132.90	114.60	-13.77
		900	12	500	360			50	132.90	112.80	-15.12
	and Eucalyptus globulus L.	900	12	500	430	-	ISO 13061-3 (2013)	50	132.90	87.10	-34.46
	neartwood	900	35	500	290			50	132.90	131.30	-1.20
		900	35	500	360			50	132.90	131.80	-0.83
		900	35	500	430			50	132.90	107.80	-18.89

Research	Wood Species	Density (kg/m <sup>3</sup> )	Initial MC (%)	MW Power (W)	Applied Energy (MJ/m <sup>3</sup> )	MW Exposure Time (s)	Flexural Testing Standard	Number of Replicates	MOR before MW Treatment (MPa)	MOR after MW Treatment (MPa)	Δ <sub>MOR</sub> (%)
Kol and Çayır [9]		472	55	925	1156	1800	- TS 2474 (1976)	84	58.05	58.68	+1.09
	Oriental spruce	472	55	1850	1156	900		84	58.05	59.16	+1.91
	(Piceu orientutis (L.) LITK.) sapwood	472	83	925	1542	2400		84	58.05	54.51	-6.10
		472	83	1850	1542	1200		84	58.05	56.05	-3.45
Kol and Çayır [18]	Oriental spruce	472	135	925	2158	3700		84	58.05	52.60	-9.39
	(Picea orientalis (L.) Link.)	472	136	1295	2158	2640	TS 2474	84	58.05	50.80	-12.49
	sapwood	472	133	1850	2158	1850	(1)/0)	84	58.05	52.30	-9.91

**Table 3.** Group C—Wood properties, MW parameters, and MOR variation values from the literature of small clear specimens, including the product of MW power by exposure time.

Research	Wood Species	Density (kg/m <sup>3</sup> )	Initial MC (%)	MW Power (W)	MW Ex- posure Time (s)	MW Power × MW Exposure Time (W.s)	Flexural Testing Standard	Number of Repli- cates	MOR before MW Treatment (MPa)	MOR after MW Treatment (MPa)	Δ <sub>MOR</sub> (%)
Hong-Hai et al. [36]		500	24.5	5000/ 9000	20/20	$2.80  imes 10^5$	-	-	85.71	97.55	97.55 +13.81
	Larch wood ( <i>Larix Olgensis</i> )	500	24.5	5000/ 9000	20/30	$3.70  imes 10^5$	-	-	96.63	96.45	-0.19
		500	24.5	5000/ 9000	30/30	$4.20  imes 10^5$	-	-	132.99	109.33	+17.79
		500	24.5	5000/ 9000	30/50	$6.00  imes 10^5$	-	-	116.36	126.41	+8.64

# Table 2. Cont.

Research	Wood Species	Density (kg/m³)	Initial MC (%)	MW Power (W)	MW Ex- posure Time (s)	MW Power × MW Exposure Time (W.s)	Flexural Testing Standard	Number of Repli- cates	MOR before MW Treatment (MPa)	MOR after MW Treatment (MPa)	Δ <sub>MOR</sub> (%)
Koiš et al. [37]	Norway spruce (Picea abies)	455	73	3000	150	$4.50  imes 10^5$	Three-Point Bending Test	-	74.00	73.00	-1.33
		370	40	14,000	60	$8.40  imes 10^5$		12	49.94	54.11	+8.35
		370	40	17,000	60	$1.02  imes 10^6$	_	12	49.94	42.64	-14.62
He et al. [24]		370	40	20,000	60	$1.20  imes 10^6$	_	12	49.94	42.74	-14.42
	China fir	370	40	20,000	30	$6.00  imes 10^5$	GB/T 1936.1 (2009)	12	49.94	48.83	-2.22
		370	40	20,000	60	$1.20  imes 10^6$		12	49.94	41.82	-16.26
	(Cullingiania anecolata)	370	40	20,000	90	$1.80  imes 10^6$	(2009)	)09) 12	49.94	37.34	-25.23
		370	30	20,000	60	$1.20  imes 10^6$	_	12	49.94	45.88	-8.12
		370	50	20,000	60	$1.20  imes 10^6$	_	12	49.94	42.47	MOR after MW Treatment (MPa) $\Delta_{MOR}$ (%)73.00-1.3354.11+8.3542.64-14.6242.74-14.4248.83-2.2241.82-16.2637.34-25.2345.88-8.1242.47-14.9640.04-19.8258.90 *-52.6455.74 *-55.1850.6-28.43
		370	70	20,000	60	$1.20  imes 10^6$	_	12	49.94	40.04	-19.82
Dorros et al [27]	Jequitibá-rosa	650	65	900	90	$8.10 imes10^4$	ASTM	20	124.37	58.90 *	-52.64
Peres et al. [27]	(Cariniana sp)	650	68	900	90	$8.10  imes 10^4$	- D143-94 (2014)	20	124.37	55.74 *	-55.18
Ouertani et al. [4]	Jack pine (Pinus banksiana)	506	36	1000	360	$3.60  imes 10^5$	ASTM D143-94 (2000)	20	70.70	50.6	-28.43

Table 3. Cont.

\* The values were adjusted to 12% since the authors did not provide them with a 12% moisture content.



Figure 1. Percentage of softwood and hardwood within the studied wood species.

#### 2.2. Pearson's Correlation Coefficient

In the first part of the analysis, Pearson's correlation coefficient (*r*) was used. It is a common approach for statistical analysis that measures the degree of correlation between two quantitative variables. This approach typically uses datasets with a normal distribution and an interval or continuous scale [34]. It can range from -1 to +1, presenting a low, moderate, or strong correlation, where a 0 value means that there is no correlation. Pearson's correlation coefficient does not only measure the linear relationships [38]; in this paper, Pearson's correlation analysis was used to reveal the relationship between the studied parameters and  $\Delta_{MOR}$ .

#### 2.3. Regression Model

Besides correlation analysis, analyses of regression models were required. A multiple linear regression model is a statistical method for examining the associations between two or more independent variables and a dependent variable [29]. In regression models, the parameters are adjusted by the least squares method.

When analyzing the regression models, the R-squared ( $\mathbb{R}^2$ ) is a statistical measure to evaluate how close the data are to the fitted regression line. It is also known as the multiple determination coefficient for multiple regression. The  $\mathbb{R}^2$  is always between 0 and 1, and the closer to 1, the better the regression model explains all the variation in the response variable around its mean. In this work, together with Pareto's charts, regression models were used to analyze the combined effect of the parameters on  $\Delta_{MOR}$ .

#### 2.4. Pareto's Charts

Pareto's charts were used to analyze the combined influence of each analyzed parameter in the variation of MOR values of MW-treated wood samples. A Pareto chart is a visual representation of the process variables and their interactions, ranked from the most to the least influential.

Pareto's chart of the standardized effects enables one to determine the most essential factors and interaction effects for the process or design optimization study at hand [39]. The influencing and non-influencing ones are separated by a threshold (a reference line) [40], indicating which parameters are statistically significant. This threshold for statistical significance depends on the significance level, which was 95%. Then, every parameter placed on the left of the threshold was not statistically significant, i.e., the bars that crossed the reference line were statistically significant at the significance level of 95%.

#### 2.5. Statistical Analysis

Analysis of variance, at a 5% significance level, was used to verify the variables' significance. In an ANOVA formulation, a *p*-value less than the significance level implies that the variable is considered significant and not significant otherwise (*p*-value > 0.05). The systematic analysis of variance (ANOVA) allows for verifying whether the terms (variables) of the regression model and the model itself are significant and the order of significance

of the terms. All the statistical analysis, Pearson's correlation coefficient, and regression models were done using Minitab software.

#### 3. Results

As explained earlier, the variables to be analyzed were the wood density and IMC, applied energy, MW Power × MW exposure time, and  $\Delta_{MOR}$ . First, the correlation analyses of the variables for Group A were carried out, as shown in Figure 2. Since the correlation of  $\Delta_{MOR}$  (Variation of MOR) with the other variables is relevant to this study, only their R<sup>2</sup>, Pearson's correlation coefficient, and *p*-values were examined. It is important to recall that these results only explain the correlation between every two variables without analyzing them combined with other parameters.



**Figure 2.** Correlation of a linear regression analysis of (**a**) density and Variation of MOR; (**b**) IMC and Variation of MOR; (**c**) applied energy and Variation of MOR for Group A.

We noticed that the isolated parameters could not explain the variations that the MOR values,  $\Delta_{MOR}$ , of structural size specimens had after MW treatment. Although the correlation analysis between applied energy and variation of MOR had a Pearson's correlation coefficient of r = 0.719, close to 1, the *p*-value was bigger than 0.05, which indicated no significance.

Figure 3 shows the correlation and ANOVA analysis for works from Group B with small clear wood specimens. As with Group A, Group B's results showed that the isolated variables could not explain the variations of MOR values after MW treatment.



**Figure 3.** Correlation of a linear regression analysis of (**a**) density and Variation of MOR; (**b**) IMC and Variation of MOR; (**c**) applied energy and Variation of MOR for Group B.

Finally, Figure 4 presents the results of correlation and ANOVA analysis for the works of Group C. Although the correlations between the variation of MOR and density and IMC were significant (p-value < 0.05), their Pearson's correlation coefficients indicated a moderate correlation.



**Figure 4.** Correlation of a linear regression analysis of (**a**) density and Variation of MOR; (**b**) IMC and Variation of MOR; (**c**) applied energy and Variation of MOR for Group C.

These first results for Groups A, B, and C indicated that the variables, when analyzed separately, could not explain the variations that occurred in the MOR of the samples after MW treatment,  $\Delta_{MOR}$ . Therefore, it was necessary to assess the effects that the combination of these parameters had on the  $\Delta_{MOR}$ . Figures 5–7 show Pareto's chart for a regression model analysis of Groups A, B and C, respectively.



Figure 5. Pareto chart for regression model analysis of Group A.



Figure 6. Pareto chart for regression model analysis of Group B.



Figure 7. Pareto chart for regression model analysis of Group C.

From ANOVA analyses, all the studied parameters were statistically significant for their respective Groups A, B, and C. For Group A, density, IMC, and applied energy *p*-values were 0.016, 0.011, and 0.003. Analyzing Figure 5, the most prominent effect in the variation of MOR values of the MW-treated structural wood specimens was applied energy, followed by IMC and density.

In the case of the works of Group B, density, IMC, and applied energy had *p*-values of 0.000, 0.002, and 0.001. Analyzing Figure 6, density was the parameter that most influenced the MOR values of MW-treated small clear wood specimens, followed by applied energy and IMC.

For the works present in Group C, density, IMC, and MW Power × MW exposure time had *p*-value equal to 0.002, 0.035, and 0.014. Figure 7 shows that density was the parameter that most affected the  $\Delta_{MOR}$  of the studied small clear wood samples, followed by the product of MW power by MW exposure time and then by the IMC.

Finally, it was verified that density was the most important parameter for the small clear specimens. In contrast, the applied energy was the most significant for the specimens with structural dimensions. The hypothesis for this lies in the fact that small clear specimens, by definition, strictly cannot contain knots, fiber deviations, and other defects, while in structural elements these defects can occur.

## 4. Discussion

Once all the studied variables (wood density and IMC, applied energy, and MW Power  $\times$  MW exposure time) were considered significant when analyzed combined to explain the  $\Delta_{MOR}$ , we started by pointing out and explaining the role that each one of them plays in wood properties and MW modification of wood samples, namely in  $\Delta_{MOR}$ .

One of the most crucial properties of wood that affects other properties is density [12]. The structure of the wood determines wood density. Density increases in softwoods as the proportion of cells with thick cell walls grows. Density in hardwoods is determined not only by the thickness of the fiber wall but also by the quantity of vacant space occupied by vessels and parenchyma. In general, "density is related to the proportion of the volume of cell wall material to the volume of lumina of those cells in a given bulk volume". The lumen lacks structure since it is the vacuum space in the cell's interior. Therefore, wood is a material with two basic domains: air space (mainly in the lumina of the cells) and the component cell walls [41].

It is important to highlight two facts. First, during MW treatment, water and steam tend to come out of the wood under high pressure, which ends up breaking cells and damaging the wood's microstructure. Weng et al. [10] explain that during the treatment, the MW energy rapidly vaporizes the water in the wood, and the increasing steam pressure causes varying degrees of damage to each wood cell tissue. Permeability and fluid flow within the wood microstructure are primarily increased by rupturing weak anatomical microstructures such as ray cells, parenchyma, and tyloses. Even the primary cell walls might be damaged [10,42].

Second, the more MW energy is supplied to the wood, the greater the damage. By using scanning electron microscopy (SEM) analysis, studies [10,42,43] showed that increasing MW energy led to an increase in damage to wood microstructure. When high MW energies were applied, many voids were created in the wood, altering several properties including porosity, permeability, strength, and flexibility [12]. In fact, experimental studies have observed that the density of MW-treated samples tended to be smaller when compared to those with no MW treatment [7,12,44].

According to Torgovnikov and Vinden [12], the number of cavities, their diameters, and distribution are highly dependent on the wood structure and may be adjusted by varying the MW modification intensity. Xiao et al. [45] also explain that the degree of structural damage is, therefore, determined by the amount of internal pressure in the wood during MW treatment. In turn, the generated steam pressure is directly linked to the MW parameters, such as power, time, and applied energy.

Torgovnikov and Vinden [12] demonstrated that, under the same MW frequency, with the same initial MC and same dimensions, hardwood specimens required more MW energy than softwoods to achieve similar levels of MW modifications. Analyzing wood densities and the quantity of applied energy of Group A (Table 1) and the results of Figure 5, the statement made by Torgovnikov and Vinden [12] can be exemplified. The radiata pine samples, a softwood with a density of 420 kg/m<sup>3</sup> and under an MW applied energy of 370 MJ/m<sup>3</sup>, presented a reduction in MOR values of around 56%. In contrast, the blue gum, a hardwood with a density of 698 kg/m<sup>3</sup> subjected to an MW applied energy of 1030 MJ/m<sup>3</sup>, had a very close reduction in MOR to MW-treated samples of radiata pine:

around 60%. In other words, blue gum specimens, which had a density 1.7 times higher than the radiata pine samples, needed 2.8 times more MW energy to have the same reduction in MOR that the radiata pine specimens had.

When analyzing the data presented in Table 2 and the results of Figure 6 for Group B, it was noticed that the wood samples were either made only of heartwood or sapwood. Sapwood samples required higher amounts of MW applied energy than the heartwood ones to cause a significant loss in the MOR values of MW-treated samples. For instance, the MW energy required to cause severe reductions in the MOR values of MW-treated eucalyptus heartwood samples was 3.7 times smaller than oriental spruce, highlighting that eucalypt density was 2.1 times higher than spruce samples (Table 2).

Torgovnikov and Vinden [12] explained that under the same level of MW applied energy, for instance, to achieve a moderate degree of modification, samples containing heartwood lost more of their strength when compared to samples with only sapwood, therefore resulting in a minor modification of MOR. Furthermore, drying wood samples with only sapwood and only heartwood under the same MW parameters, Mascarenhas et al. [16] showed that small clear wood specimens of Portuguese maritime pine (*Pinus pinaster*) containing only heartwood had a significant improvement in water uptake when compared to the samples containing only sapwood, i.e., to cause a significant improvement in water uptake it would be necessary to supply the sapwood sample with more MW energy. Therefore, based on these analyses, the density of the wood element was, in fact, a significant parameter (that influences) the variations in the MOR values after MW treatment.

Concerning the analysis of the IMC, we can understand the following. The water is present in wood in two ways, "as free water (liquid water or water vapor in cell lumina and cavities) or as bound water (held by intermolecular attraction within cell walls)". In this context, the concept of fiber saturation point (FSP) is important. It is defined as the point where "only the cell walls are completely saturated (all bound water), but no water exists in cell lumina" [41]. The FSP usually is around 30%. Although the FPS is conceptually straightforward, in practice, measuring the exact split of "free" and "bound" water is complex, i.e., the FSP does not exist in real wood species as it is a theoretical state [41]. Under the FSP, wood's physical and mechanical properties tend to vary depending on the MC [46]. Mvondo et al. [47] also remarked that from the anhydrous point to the FSP, MC highly influences the wood's elastic characteristics and yield strength.

Due to the type of interaction that bound water has and the 'place' where it is in wood cells, it can only be eliminated with the rupture of the molecular structure of the wood. Free water is rapidly eliminated when the living tree is cut until it reaches the average moisture content of around 30%, the FSP [48]. Among the wood species analyzed for the regression model and Parato's analyses (their combined effects), their IMC ranged from 12 to 164%. Based on these reference values, the results demonstrated that higher initial MC corresponded with slight reductions in the MOR of MW-treated samples.

Under high IMC, most MW energy was used to heat and evaporate the free water during the MW treatment. Assuming that more energy had been used for this purpose, less damage to the cellular structures of the wood might have happened when compared to the MW drying process in which the wood specimens had initial MC values lower than 30%. Sethy et al. [49] explained that more energy was required to dry wood below the FSP, and this energy needed to be raised as the moisture content fell. As the amount of energy required to dry increased, the more significant the reductions in flexural strength were.

When examining the results presented by Hermoso and Vega [13] (Group B), the authors observed that the MOR values of MW-treated wood samples with an IMC of 12% were smaller than those with an initial MC above 30%. They used three different MW applied energies; for all of them, the MOR values of dried samples were statistically different from the control group. In the case of samples with IMC higher than 30%, a difference was noticed only under the higher MW applied energy compared to the control samples.

Wang et al. [26] pointed out that using MW intensity (applied energy) ranging from 72 MJ/m<sup>3</sup> to 288 MJ/m<sup>3</sup>, the cell wall morphology did not change noticeably for any of the samples with IMC of 40%, and the cell structures were unaltered. On the other hand, the MW treatments with applied energy of 288 MJ/m<sup>3</sup> in wood samples with an IMC of 20% produced the worst modifications in the cell wall structure. Hence, the damage produced in the cell morphology was directly linked to the decrease in MOR values of MW-treated wood samples.

Using samples of *Pinus radiata* with an average IMC of 35% and *Eucalyptus obliqua* with an average IMC of 91% with the same dimensions, treated with the same MW frequency and moderate applied energy, Torgovnikov and Vinden [12] (Group A) showed that the wood specimens exhibited different levels of reduction in the MOR values. *Pinus radiata* reduced from 4 to 26% in MOE and MOR, and *Eucalyptus obliqua* had a 12 to 17% reduction in MOR. The same happened with *Eucalyptus globulus*, with an IMC of 98%, and *Paulownia fortune*, with an initial MC of 164%. *Eucalyptus globulus* had a decrease of 60% in MOR, and *Paulownia fortune* had a decrease of 45% in MOR.

Acknowledging the data from Table 3 and the results presented in Figure 6 (Group C), we can analyze the results from He et al. [24]. Since all the wood samples used by the authors had an IMC above the FPS (higher than 40%), they explained that no significant impact was measured in the MOR values of MW-treated samples under the MW treatment conditions used by them. Then, for these cases, the higher the initial MC, the smaller the decrease in MOR in static bending tended to be.

Finally, the MW applied energy *E* (Groups A and B) and the MW Power  $\times$  MW exposure time (Group C) were significant, presenting high importance in the variation of MOR values. The higher the MW applied energy and the MW power and exposure time, the higher the reduction in MOR values in bending of MW-treated specimens tended to be. In fact, this has been proved by experimental works in the literature. High MW intensity, which can be understood either as the MW applied energy or as the product of MW power per exposure time, led to higher modifications in the microstructure and, therefore, a considerable reduction in MOR [12,42].

For instance, under the same IMC, increasing the applied energy from 320 to 342 MJ/m<sup>3</sup>, the variations in the MOR of Red Stringybark went from +2.7 to -10.8%. Hermoso and Vega [10] increased the applied energy by 32.6% and identified a reduction of 31.6% in the MOR of MW-treated samples. Other data from the literature that support our findings were presented by He et al. [32]. By increasing the MW Power × MW exposure time from  $6.00 \times 10^5$  to  $1.20 \times 10^6$  W.s, MOR values reduced from -2.2 to -14.4% [32].

Ganguly et al. [42] showed that enhanced MW treatment exposure reduced skeletal density. Moreover, with increasing MW treatment, the specific pore volume and percentage of porosity increased. The authors also stated that SEM images allowed the observing of deformities in the earlywood tissue structure in treated heartwood samples under high MW treatment intensity.

Weng et al. [50] used two different MW powers, 15 and 20 kW. They showed that under high-intensity MW treatment (20 kW), the damage to the wood microstructure was more severe than after low-intensity MW treatment (15 kW). The formed macro-cracks ranged in width from 100 to 130  $\mu$ m for 20 kW and from 1 to 25  $\mu$ m for 15 kW. The diameter of pores of the margo capillaries in pit membranes increased by 23% for 15 kW and 55% for 20 kW. Besides previous works, both results presented by Ganguly et al. [42] and Weng et al. [50] evidenced and supported the results of this study, showing that the effect caused by the applied energy in the density directly impacted the MOR values.

Finally, analyzing the results provided by Torgovnikov and Vinden [12], who MWtreated four different wood species, we can briefly exemplify and summarize what was previously discussed. When examining the density in isolation, it was not possible to reach a clear conclusion to explain the reductions in MOR found by the authors for the four wood species, just as it was not possible to explain the isolated effects of IMC and applied energy in the diminishing of MOR after MW modification. However, the understanding and explanation of the reductions in MOR values after MW treatment were feasible by investigating the combination of these parameters [12].

Thus, even with the reduced database, there were works present in the literature with sufficient information about MW treatment and the experimental investigation of the impact on MOR. It was possible to obtain significant results regarding the influences each analyzed variable had on MOR, considering not only their isolated effect but, above all, the combined effect through the analysis of their *p*-values. Although some experimental works present in the literature demonstrated the influence of MW applied energy, for example, this work is the first that uses different databases to, based on statistical methodologies, demonstrate and explain the impact of these variables in the variations of MOR in the bending of MW-treated samples.

The results and analysis made above showed that when separately analyzing wood density, IMC, MW energy applied, and MW Power × MW exposure time, it was not possible to quantify and understand the impact each of them has on  $\Delta_{MOR}$ . However, since these parameters are interconnected, an analysis of their combined impact was necessary to clearly understand the relevance that each one had in the overall modification of MOR,  $\Delta_{MOR}$ . Thus, when analyzed together, wood density, IMC, MW energy applied, and MW Power × MW exposure time could explain  $\Delta_{MOR}$ .

Hence, even in the face of the growing number of research works investigating the use of MW technology to increase the permeability of wood species, there is still a need to conduct research with different species aiming to investigate the improvements in the permeability [11], and especially verify the effects on the mechanical properties of small clear and structural size specimens. In this sense, this work presented results that can guide future investigations.

#### 5. Conclusions

From the 11 works of the literature used in this paper, it was possible to obtain 37 different MW treatment programs with relevant and sufficient data to be used. The very first parameters chosen to be investigated and their influences on  $\Delta_{MOR}$  values in bending were wood density and IMC, samples' dimensions, MW power, applied energy, exposure time, and frequency. After passing through a selective process of choosing the variables to be studied, they were placed and investigated in three groups of works from the literature. Group A contained works that had information about wood density, IMC, and MW energy applied for wood samples with structural size. Group B contained works that had information about wood density, IMC, and MW energy applied for small clear wood specimens. Group C contained works that had information about wood density, IMC, and MW Power × MW exposure time for small clear specimens.

The results and analysis made in this research work revealed that the isolated effects of the studied variables could not be used to explain the  $\Delta_{MOR}$ . Nevertheless, the combined effect of wood density, IMC, MW energy applied, and MW Power × MW exposure time could be used to quantify and explain the reductions that the MOR of MW-treated specimens had by carrying out an analysis of variance, a correlation analysis, and a statistical regression models analysis.

Finally, despite the limited database of publications in the literature with relevant information about MW treatment and the experimental study of its impact on MOR, important results were highlighted. They were capable of addressing the questions raised in the objectives. Thus, researchers interested in investigating MW-treated wood species that have not yet been studied regarding the effects on MOR should pay special attention to those parameters. Complementarily, the outcomes of this work, associated with new experimental studies with other wood species, can support the development of mathematical models for predicting the MOR of MW-treated wood specimens.

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